PYXIS REGULATORY CONSULTING

11324 17th Ave. Ct. NW
Gig Harbor, WA 98332

Phone: 253-853-7369
Fax: 253-853-5516
Email: JLW@nwrain.com

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13 pp

December 21, 1999

MR 3005

PRIORITY MAIL

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Information Management Division
Office of Pollution Prevention and Toxics
Environmental Protection Agency
Room G009
401 M Street, S.W.
Washington, D.C. 20460

30375

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1999 DEC 27 AM 11: 35

ATTN: Gwen Shepard

RE: Submission of sanitized protocol

Dear Gwen:

Pursuant to our telephone conversation, this copy of the protocol **replaces** the sanitized copy previously submitted November 15, 1999. This does not constitute a resubmission of the protocol, but a submission to comply with Confidential Business Information requirements. A new protocol will be submitted shortly that incorporates the requested changes previously discussed.

Please feel free to contact me if you have any questions or need any additional information.

Sincerely,

Janelle Whitehouse

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Enclosures

cc: Stuart Feldstein; Albaugh, Inc.
Glen O'Brien, Albaugh, Inc.

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COMPANY SANITIZED

November 15, 1999

OVERNIGHT DELIVERY

Document Control Office
Mail Code 7407
Information Management Division
Office of Pollution Prevention and Toxics
Environmental Protection Agency
Room G009
401 M Street, S.W.
Washington, D.C. 20460

MP 30376

ATTN: Dioxin/Furan Report

RE: Submission of draft protocol for the testing of [Material Redacted]

Dear Sir or Madam:

Pursuant to 40 C.F.R. §§ 766.35(a)(2), 790.50 and Section 4 of TSCA, Albaugh, Inc. is submitting six (6) copies of the draft protocol for the testing of [Material Redacted], CAS Number [Material Redacted], under 40 C.F.R. Part 766.

Albaugh hereby claims business confidentiality for the identity of the test substance covered by this protocol submission; a redacted copy of this letter and accompanying protocol is enclosed for use by the EPA. Per 40 C.F.R. Part 790.7, Albaugh, Inc. claims confidentiality, indefinitely, for the following reasons:

- The test substance is the main starting material for the manufacture of [Material Redacted]. The test substance is identified in sections of the study plan.
- As there are several methods and starting materials that can be used to manufacture [Material Redacted], revealing the identity of the test substance would harm Albaugh's competitive position. Competitors could use this confidential information, to the detriment of Albaugh, to modify, or develop, a manufacturing process to that similar to Albaugh's.
- Albaugh has not revealed to the public the processes for the manufacture of [Material Redacted]. A detailed description of the process was provided to the EPA, Office of Pesticide Programs in order to register this product. In compliance with Pesticide Registration Notice 86-5, Albaugh prepared, formatted and submitted its reports to the EPA that carefully claimed all information confidential that pertained to the identity of the starting materials and manufacturing process of [Material Redacted]. Further, all individuals or companies that are provided information regarding the starting materials and manufacturing process are subject to a confidentiality agreement with Albaugh.

- To the best of Albaugh's knowledge, this information has not been disclosed in a patent.
- Finally, the USEPA – Office of Pesticide Programs considers this information confidential on the basis of the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) Section 10(d)(1)(A). A copy of the relevant section is attached to this letter.
- Albaugh acknowledges that "health and safety data" regarding the test substance as defined by 40 C.F.R. § 2.306(a)(3) are not eligible for confidential treatment.

We trust you will find this protocol is complete and in compliance with the TSCA requirements. Please feel free to contact me if you have any questions or need any additional information.

Sincerely,

Janelle Whitehouse

Enclosures

cc: Stuart Feldstein; Albaugh, Inc.
Glen O'Brien, Albaugh, Inc.

which is adulterated, misbranded, not registered (in the case of a pesticide) or otherwise in violation of this Act and in the event of the inability of any person to produce records containing such information, all other records and information relating to such delivery, movement, or holding of the pesticide or device; and

(3) the seizure of any pesticide or device which is in violation of this Act.

(c) ENFORCEMENT.—

(1) CERTIFICATION OF FACTS TO ATTORNEY GENERAL.—The examination of pesticides or devices shall be made in the Environmental Protection Agency or elsewhere as the Administrator may designate for the purpose of determining from such examinations whether they comply with the requirements of this Act. If it shall appear from any such examination that they fail to comply with the requirements of this Act, the Administrator shall cause notice to be given to the person against whom criminal or civil proceedings are contemplated. Any person so notified shall be given an opportunity to present the person's views, either orally or in writing, with regard to such contemplated proceedings, and if in the opinion of the Administrator it appears that the provisions of this Act have been violated by such person, then the Administrator shall certify the facts to the Attorney General, with a copy of the results of the analysis or the examination of such pesticide for the institution of a criminal proceeding pursuant to section 14(b) or a civil proceeding under section 14(a), when the Administrator determines that such action will be sufficient to effectuate the purposes of this Act.

(2) NOTICE NOT REQUIRED.—The notice of contemplated proceedings and opportunity to present views set forth in this subsection are not prerequisites to the institution of any proceeding by the Attorney General.

(3) WARNING NOTICES.—Nothing in this Act shall be construed as requiring the Administrator to institute proceedings for prosecution of minor violations of this Act whenever the Administrator believes that the public interest will be adequately served by a suitable written notice of warning.

SEC. 10. [136h] PROTECTION OF TRADE SECRETS AND OTHER INFORMATION.

(a) IN GENERAL.—In submitting data required by this Act, the applicant may (1) clearly mark any portions thereof which in the applicant's opinion are trade secrets or commercial or financial information and (2) submit such marked material separately from other material required to be submitted under this Act.

(b) DISCLOSURE.—Notwithstanding any other provision of this Act and subject to the limitations in subsections (d) and (e) of this section, the Administrator shall not make public information which in the Administrator's judgment contains or relates to trade secrets or commercial or financial information obtained from a person and privileged or confidential, except that, when necessary to carry out the provisions of this Act, information relating to formulas of products acquired by authorization of this Act may be revealed to any

Federal agency consulted and
or in findings of fact issued by

(c) DISPUTES.—If the Adm
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be protected from disclosure
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for inspection such data until
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(d) LIMITATIONS.—

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Federal agency consulted and may be revealed at a public hearing or in findings of fact issued by the Administrator.

(c) DISPUTES.—If the Administrator proposes to release for inspection information which the applicant or registrant believes to be protected from disclosure under subsection (b), the Administrator shall notify the applicant or registrant, in writing, by certified mail. The Administrator shall not thereafter make available for inspection such data until thirty days after receipt of the notice by the applicant or registrant. During this period, the applicant or registrant may institute an action in an appropriate district court for a declaratory judgment as to whether such information is subject to protection under subsection (b).

(d) LIMITATIONS.—

(1) All information concerning the objectives, methodology, results, or significance of any test or experiment performed on or with a registered or previously registered pesticide or its separate ingredients, impurities, or degradation products, and any information concerning the effects of such pesticide on any organism or the behavior of such pesticide in the environment, including, but not limited to, data on safety to fish and wildlife, humans and other mammals, plants, animals, and soil, and studies on persistence, translocation and fate in the environment, and metabolism, shall be available for disclosure to the public. The use of such data for any registration purpose shall be governed by section 3 of this Act. This paragraph does not authorize the disclosure of any information that—

(A) discloses manufacturing or quality control processes,

(B) discloses the details of any methods for testing, detecting, or measuring the quantity of any deliberately added inert ingredient of a pesticide, or

(C) discloses the identity or percentage quantity of any deliberately added inert ingredient of a pesticide,

unless the Administrator has first determined that disclosure is necessary to protect against an unreasonable risk of injury to health or the environment.

(2) Information concerning production, distribution, sale, or inventories of a pesticide that is otherwise entitled to confidential treatment under subsection (b) of this section may be publicly disclosed in connection with a public proceeding to determine whether a pesticide, or any ingredient of a pesticide, causes unreasonable adverse effects on health or the environment, if the Administrator determines that such disclosure is necessary in the public interest.

(3) If the Administrator proposes to disclose information described in clause (A), (B), or (C) of paragraph (1) or in paragraph (2) of this subsection, the Administrator shall notify by certified mail the submitter of such information of the intent to release such information. The Administrator may not release such information, without the submitter's consent, until thirty days after the submitter has been furnished such notice. Where the Administrator finds that disclosure of information described in clause (A), (B), or (C) of paragraph (1) of this subsection is necessary to avoid or lessen an imminent and sub-

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Protocol

[Material Redacted]:
Dibenzo-Para-Dioxins/Dibenzofurans Testing

Data Requirements

40 C.F.R. § Part 766 Dibenzo-Para-Dioxins/Dibenzofurans

Testing Facility

Quanterra, Inc
880 Riverside Parkway
West Sacramento, California 95605
Telephone: (916) 373-5600; Facsimile: (916) 372-1059

Sponsor

40000000008

Albaugh, Inc.
121 NE 18th Street
Ankeny, IA 50021
Telephone: (515) 964-9444; Facsimile: (515) 964-7813

Protocol Approval/Acceptance:
For Quanterra Inc.

_____ _____
[Name – to be determined] Date
Study Director

For Albaugh, Inc.

_____ _____
Janelle Whitehouse Date
Agent

Table of Contents

1.0 Project Description

To satisfy requirements for testing chemical substances that may be contaminated with halogenated dibenzodioxins (HDDs)/dibenzofurans as defined in section 4 of the Toxic Substances Control Act (TSCA), 15 U.S.C. 2603 and 40 C.F.R. § 766.3 and requirements for reporting under section 8 of TSCA, 15 U.S.C. 2607. The testing requirements for which Albaugh intends to conduct tests are all requirements set forth in 40 C.F.R. § 766 applicable to [Material Redacted].

Study Number: To be determined

Personnel: To be determined

Study Director: To be determined

Proposed Schedule:

Estimated Experimental Start Date: June 1, 2000
Estimated Experimental Termination Date: July 1, 2000

Test Substance

Name: [Material Redacted]

Albaugh, Inc. imports [Material Redacted] from two separate sources. Both sources utilize the same manufacturing process. Therefore, Albaugh, Inc. will obtain samples for analysis from the source with the greatest volume of sales, which, in this instance, also has the greatest potential for exposure to intense heat and alkalinity during the manufacturing process.

2.0 Project Organization and Management

Quanterra Inc. will perform and manage the project under the supervisions of Albaugh, Inc.

3.0 Personnel Qualifications

The testing facility management of Quanterra Inc. will ensure only properly trained and proficient personnel will handle test material and perform the analyses. Qualifications of personnel are documented by curriculum vitae that include academic history, employment history, experience, and memberships in professional organizations.

Quanterra personnel receive internal, external, formal, and informal training. Training is performed to maintain and develop proficiency, and to promote improvement. Training is performed by individuals knowledgeable in the subject matter. Personnel are qualified by their experience and training and are only assigned duties for which they are qualified.

4.0 Facilities, Equipment, Consumables, and Services

4.1 Facilities and Equipment

4.1.1 Evaluation

Prior to the initiation of the study, or concurrent with a critical phase audit, the sponsor or sponsor's representative will conduct an evaluation of Quanterra Inc.'s facilities to ensure compliance with TSCA GLP guidelines.

4.1.2 Inspections and Maintenance

Quanterra Inc. will maintain adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of stable power. The maintenance and use of Quanterra Inc.'s facilities and proper operations are described in Quanterra's Chemical Hygiene Plan (CHP).

The Laboratory Manager will ensure a properly maintained facility and that samples are stored properly without contamination, work areas are equipped with adequate bench, hood and operational space, and the areas are free from chemical and radiological contamination that may affect analytical results.

Frequency of preventive maintenance along with recommended preventative maintenance schedules are provided in Quanterra's Quality Assurance Management Plan (QAMP), available from Quanterra Inc. upon request, for analytical instrumentation and equipment or defined in operation specific routine maintenance Standard Operating Procedures (SOPs). Frequency of maintenance for the facility systems is documented in Quanterra's Chemical Hygiene Plan (CHP).

4.1.3 Calibration Procedures

All instruments and equipment used at Quanterra operations are controlled by a formal calibration process.

High-Resolution Concentration Calibration Solutions – Five tetradecane solutions containing 17 unlabeled and carbon-labeled (totaling 11) PCDDs and PCDFs at known concentrations are used to calibrate the instrument in accordance with Quanterra's SOP No. SAC-ID-005: Method 8290 – Polychlorinated Dioxins & Furans by HRGC/HRMS, Revision No. 4.0, Section 10 that may be found in Attachment 1. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/µL) and the highest for the octachlorinated congeners (2000 pg/ µL).

Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

4.2 Consumables and Services

Quanterra Inc. will ensure that all materials, instruments, reagents, and other purchases are of high quality and fit for high-resolution testing. The process for the procurement of items and services is described in Quanterra's Quality Assurance Management Plan (QAMP).

5.0 Data Generation

5.1 Sample Collection

The manufacturer will ensure sample containers are prepared and samples collected in accordance with Section 8.0 of the Guidelines for the Determination of Halogenated Dibenzo-*p*-dioxins and Dibenzofurans in Commercial Products. Samples will be randomly selected from seven separate batches of [Material Redacted]. A random number table will be utilized to determine sampling times. Locations of sample collection sites will be listed and chosen through similar random selection procedures. Samples will be collected in a manner that ensures adequate representation while avoiding cross-contamination. Samples will be stored in a cool, dry area protected from light.

Documentation of the sampling plan, sample collection procedures and traceability records, including any deviations will be maintained by the manufacturer. At sampling, the following data will be recorded:

- Dates and times of sample collection and chemical analysis of samples
- Exact location and time of sample collection
- Process or product batch or lot identification
- Traceability records employed in the sample collection and ultimate location of the samples.

A maximum of seven 100 g samples of [Material Redacted] will be provided to Quanterra, Inc. for analysis.

5.2 Sample Custody

Samples shall be received and logged in by a designated sample custodian or other trained personnel. Upon sample receipt, the sample custodian shall, as appropriate:

1) Wear appropriate personal protective equipment. At a minimum, this consists of gloves, a lab coat, safety glasses, and in some cases a respirator
2) Examine the shipping containers to verify that the custody tape is intact
3) Examine all sample containers for damage

4) Open shipping containers in adequately ventilated areas to assure worker safety
5) Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the chain-of-custody
6) Compare samples received against those listed on the chain-of-custody
7) Verify that sample holding times have not been exceeded
8) Examine all shipping records for accuracy and completeness
9) Determine sample pH (if required for the scheduled analysis) and record on the chain-of-custody
10) Sign and date the chain-of-custody immediately (only after shipment is accepted) and attach the waybill
11) Note any problems associated with the coolers and samples on the chain-of-custody and immediately initiate a Condition Upon Receipt Report (CUR) or equivalent format, and notify the Project Manager who in turn notifies the client
12) Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
13) Place the samples in proper laboratory storage and notify appropriate personnel

A chain-of-custody form will be utilized which will include: Name of individual removing the sample, date sample was removed and returned, identification of tests to be performed, sample matrix, laboratory sample number and sample storage location.

5.3 Test Method

Analytical test methods will follow those developed in Guidelines for the Determination of Polyhalogenated Dibenzo-*p*-dioxins and Dibenzofurans in Commercial Products pursuant to 40 C.F.R. § 766.12 and necessary modifications of Section 11 of Quanterra SOP No. SAC-ID-0005, Revision No. 4.0 (Attachment 1).

Exposure to chemicals will be maintained as low as reasonably achievable. Unless chemicals are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Standards and reagents will be prepared in a fume hood with the sash closed as far as the operation will permit.

Chemical extraction and clean up procedures will follow those developed in Guidelines for the Determination of Polyhalogenated Dibenzo-*p*-dioxins and Dibenzofurans in Commercial Products pursuant to 40 C.F.R. § 766.12 and necessary modifications of Section 9.3 of SOP No. SAC-ID-0005, Revision No. 4.0 (Attachment 1).

Instrumentation utilized will meet the recommendations set forth in Guidelines for the Determination of Polyhalogenated Dibenzo-*p*-dioxins and Dibenzofurans in Commercial Products pursuant to 40 C.F.R. § 766.12 and necessary modifications of Section 6 of SOP No. SAC-ID-0005, Revision No. 4.0 (Attachment 1).

Recommended GC operating conditions utilizing a 60-m DB-5 fused-silica capillary column are as follows:

Injector Temperature: 280°C
Interface Temperature: 280°C
Initial Temperature and Time: 190°C/1 Minute
Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last of the tetra-group has eluted from the column. The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

Control samples and HDD/HDF-reinforced control samples, isotopically labeled compounds, and duplicate samples will be handled according to procedures set forth Section 9 of SOP No. SAC-ID-005, Revision No. 4.0 (Attachment 1) and may be modified as necessary.

5.4 Quality Control

Quality control procedures to be followed may be found in Section 9 of SOP No. SAC-ID-0005, Revision No. 4.0 (Attachment 1) and may be modified as necessary.

5.5 Performance and System Audits

Prior to initiation of the study or concurrent with a critical phase audit, the sponsor or sponsor's representative will ensure that all performace and system audits are performed in accordance with TSCA GLP guidelines.

6.0 Data Processing

6.1 Data Collection

Data generated during the conduct of the study will be promptly and accurately recorded in ink. All data entries will be dated on the day of entry and signed or initialed by the individual making the entry. Any change in entries will indicate the reason for the change and be dated and signed or initialed by the individual making the change.

6.2 Data Reduction and Verification

Data review procedures, defined as a set of computerized and manual checks applied at appropriate levels of the measurement process, will be clearly defined for all measurement systems in SOPs. Calculations will be checked from the raw data to the final value prior to reporting results for each group of samples. Data

reduction will be performed by the analyst who obtained the data or by another analyst. Data verification will be performed by the analyst who will conduct a 100 percent review of the data to ensure the work was done correctly. A second reviewer will verify that data reduction has been performed correctly and that the analytical results correspond to the data acquired and processed.

Following the completion of the initial verification by the analyst performing the data reduction, a second level reviewer will examine the data signed by the analyst. This examination will include an evalution of all the items required in the raw data package.

7.0 Data Quality Assessment

7.1 Precision and Accuracy

Precision and accuracy of the test method and instrumentation utilized is described in Section 6 of SOP No. SAC-ID-0005, Revision No. 4.0 (Attachment 1) and may be modified as necessary.

7.2 Representativeness

Every effort will be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

7.3 Comparability

To ensure comparability, all analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting data.

7.4 Completeness

At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

8.0 Corrective Action

Corrective actions are measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated will be documented.

9.0 Documentation and Reporting

9.1 Retention of Records

The original raw data, final report, protocol and facility records will be archived at Quanterra, Inc. Sampling data will be stored at the manufacturer's facility; copies of which will be provided to the sponsor.

9.2 Changes to the Protocol

All protocol amendments will be documented in writing by the Study Director and will be approved by the Sponsor. The changes will be in the form of an amendment and will describe what is being changed, the reason for the change and the date of the change. The amendment will be signed by the Study Director and Sponsor and will be distributed to all holders of the original protocol. Protocol deviations will be noted in the final report.

9.3 Reporting of Study Results

The final report will be the responsibility of the Study Director and will meet the guidelines set forth in 40 C.F.R. 792.185. The report will include at least the following information:

- The name and address of the testing facility and the dates the study was initiated and completed
- Study objectives and procedures as stated in the protocol
- A statement of the statistical methods employed
- Identification of the test, control and reference substances
- A description of the stability of the test, control, and reference substances
- A description of the methods used
- A description of the test system used
- A description of all circumstances that may have affected the quality or integrity of the data
- The name of the study director, scientists or other professionals, and all supervisory personnel involved in the study
- A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data, and a statement of the conclusions drawn from the analysis
- The signed and dated reports of each of the individual scientists or other professionals involved in the study including those who may analyze or evaluate data from the study after generation is completed
- A statement certifying that testing adhered to TSCA GLPs
- The location where all raw data and final report will be stored
- The signed and dated statement by the Quality Assurance Unit specifying the dates inspections were made and findings reported
- The dated signature of the Study Director.

Attachment 1

Operation-Specific Standard Operating Procedure Title: Method 8290 - Polychlorinated Dioxins & Furans by HRGC/HRMS (SOP No. SAC-ID-0005, Revision No. 4.0)